

Citation for published version:

Khotkevych, VV & Bending, SJ 2009, 'A milliKelvin scanning Hall probe microscope for high resolution magnetic imaging', *Journal of Physics: Conference Series*, vol. 150, no. 1, 012021. <https://doi.org/10.1088/1742-6596/150/1/012021>

DOI:

[10.1088/1742-6596/150/1/012021](https://doi.org/10.1088/1742-6596/150/1/012021)

Publication date:

2009

Document Version

Peer reviewed version

[Link to publication](#)

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A milliKelvin scanning Hall probe microscope for high resolution magnetic imaging

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Abstract. The design and performance of a novel scanning Hall probe microscope for milliKelvin magnetic imaging with submicron lateral resolution is presented. The microscope head is housed in the vacuum chamber of a commercial ^3He -refrigerator and operates between room temperature and 300 mK in magnetic fields up to 10 T. Mapping of the local magnetic induction at the sample surface is performed by a micro-fabricated 2DEG Hall probe equipped with an integrated STM tip. The latter provides a reliable mechanism of surface tracking by sensing and controlling the tunnel currents. We discuss the results of tests of the system and illustrate its potential with images of suitable reference samples captured in different modes of operation.

1. Introduction

The scanning Hall probe microscope (SHPM) represents a very successful approach to high resolution magnetic imaging. It can be considered as an effective tool for the direct, quantitative and almost completely non-invasive measurement of local magnetic fields at the submicron level. The Hall sensors can be routinely fabricated with $\sim 100\text{nm}$ spatial resolution. In addition to their excellent geometrical characteristics and impressive signal/noise ratios, especially at cryogenic temperatures, high probe current densities enable operation in magnetic fields up to several Teslas.

Over more than a decade we have developed a series of scanning Hall probe microscopes [1] operating in a wide temperature range 4.2 - 300 K. In order to address some important contemporary issues in magnetic and superconducting materials in the milliKelvin temperature range we have designed a new SHPM which is capable of operation in the vacuum chamber of a ^3He -refrigerator.

2. Instrument design

2.1. Cryostat

Our primary motivation in selection of an Oxford Instruments Heliox VT-50 ^3He -refrigerator was its simple and robust design and excellent working characteristics. The Heliox VT-50 system is guaranteed to reach a 280 mK base temperature without additional heat load and maintain 350 mK under $50\text{ }\mu\text{W}$ load for more than 8 hours. It fits into a popular Oxford Instruments variable temperature insert (VTI) equipped with its own temperature controller. The VTI is used as an intermediate 1.5 K refrigeration stage inside the main Oxford Instruments Helium cryostat housing the 11 T superconducting solenoid.

In order to suppress ground-born mechanical vibrations the main Helium cryostat rests on a rigid non-magnetic anti-vibration table equipped with pneumatic mounts. Due to its large mass the cryostat has the advantage of being quite resistant to external vibrations. The flexible pumping line connecting the VTI with a rotary pump is passed through a massive concrete block.

In the course of initial tests of the microscope in STM-mode, which is most vulnerable to vibrations and acoustic noise, we found the above measures adequate. Bubbling of cryogenic liquids also did not exert a noticeable influence on the tunnel currents and imaging.

2.2. Microscope low-temperature detachable head

The basic concept of the SHPM head design is that it can be easily detached from the ^3He -refrigerator insert in order to provide good access to the Hall sensor and sample, enabling their quick and safe replacement, and also allowing fine adjustment of the sensor location with respect to the sample under an optical microscope.

Special care was taken to achieve a highly axially-symmetrical design of the head in order to prevent unacceptable temperature-induced bending of the microscope.

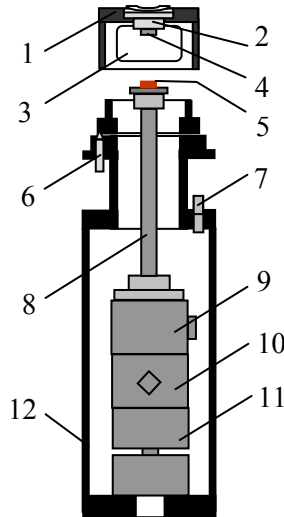


Figure 1. The SHPM head: 1 - sample holder cup, 2 - sample holder, 3 - window, 4 - sample, 5 - Hall sensor, 6 - adjustment screw, 7 - electrical connector, 8 - scanner, 9,10 - ANPx100LT positioners, 11 - ANPz100LT positioner, 12 - brass microscope body.

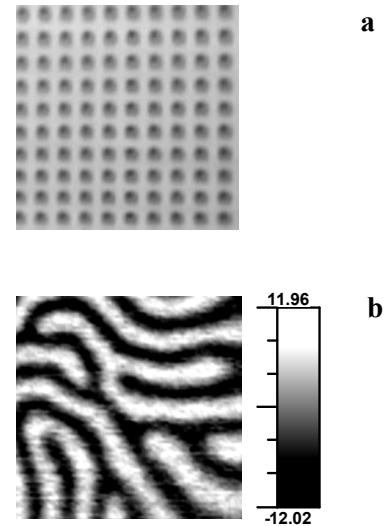


Figure 2. a - STM-image of platinum-plated 1 μm step calibration grid. b - magnetic-domain patterns in YIG - thin film, captured with scanning rate 300 $\mu\text{m/s}$. The scale is in Gauss.

The head is fitted into a cylindrical space 36 mm in diameter and 135 mm high, which is sufficient to accommodate a stack of three commercially available slip-stick nanopositioners from Attocube Systems AG for coarse three-axis positioning along with a 2 inch piezoscanner tube. These nanopositioners (two ANPx100LT for horizontal movement and one ANPz100LT for vertical motion) are guaranteed to produce controlled and highly reproducible steps from 10 to 500 nm at helium temperatures in a vacuum environment [2]. The given geometry makes it possible to exploit the entire translation range (6 mm \times 6 mm \times 7 mm) of the nanopositioners for initial adjustments of the Hall probe location. The scan range of the 2 inch piezoscanner can reach 170 μm \times 170 μm \times 4.7 μm at room temperature, when the full swing (± 400 V) of drive voltage is applied. It decreases with temperature, but nevertheless exceeds 22 μm \times 22 μm \times 0.6 μm at 4.2 K.

The microscope body is composed of a brass shell which contains the stack of nanopositioners with the scanner, electrical connectors and an easily-detachable sample-holder cup. The latter carries a 12 mm brass or copper sample-holder disk clamped with a flat bronze spring and has side windows for visual checking and adjustment of the tilt angle between the sample surface and the Hall probe chip. This angle does not usually exceed 0.5–1.0 degree, and can be aligned by means of three fine-thread adjustment screws while viewing with a binocular microscope. Samples are glued to the sample-holder disks with a thin insulating ceramic washer and can be replaced by simply sliding them through the side windows.

As in previous designs, the Hall probe (typically GaAs/AlGaAs or AlGaAs/InGaAs/GaAs 2DEG Hall sensor [1, 3] with integrated STM tunnelling tips, which can achieve minimum detectable fields $\geq 10 \text{ mG/Hz}^{1/2}$) is bonded onto a thin dielectric substrate and glued to a miniature printed circuit board with pads for Au wire bonding and two screw holes for rapid mounting. This assembly is screwed directly onto the end of the piezoscanner, which carries an array of gold-plated spring contacts to provide reliable electrical connections to the mini-board pads.

Non-magnetic materials only were used in the construction so that the microscope is capable of operation in high magnetic fields ($\leq 10\text{T}$).

2.3. Electronics

Our system is based on the commercial SPM-controller from NanoMagnetics Instruments Ltd., which provides scanning, automatic surface approach and data acquisition. Several additional modules were constructed to meet the demands of our hardware development. Firstly new high-voltage (HV) boosters with output swing $\pm 400 \text{ V}$ were built around Apex PA94 low-noise HV operational amplifiers in order to increase the scanning range 2-3 times. A custom-designed three-channel controller for the Attocube positioners was also constructed, which is triggered by the same HV pulses that the SPM-controller utilizes for the slider-motor of the commercial NanoMagnetics Instruments Ltd. SHPM and converts them into the standard Attocube positioner's waveforms. For the tunnel current and the Hall probe voltage measurements we use a homebuilt current-to-voltage converter (10^8 V/A) and a low-noise differential preamplifier with gain $G=1000$ respectively, operating at room temperature. They are mounted in a separate screened case at the top of the Heliox VT-50 insert.

2.4. Wiring, thermal anchoring and thermometry

Connection of the detachable SHPM head to the cold flange of the ^3He -pot is performed by means of the mounting tube, which is permanently bolted to the flange and plays an important role of acting as a thermal bridge to the brass microscope body. Polishing of all external surfaces of the tube and the microscope head minimizes radiation heat transfer.

We used the small-diameter UT 20-SS-SS stainless-steel coaxial cables from Microcoax for wiring the scanner electrodes and the sensors while combined lines composed of Cu joined to Nb-Ti superconducting wire were employed for the Attocube nanopositioners. All the electrical leads, which connect the microscope head to the top of the Heliox VT-50 are thermally anchored at the vacuum chamber upper flange, and then at the 1K-plate, by firmly winding them around the copper anchoring posts. This winding also creates highly efficient low-pass filters for radio-frequency pick-up suppression.

In addition to the main RuO_2 thermometer on the ^3He pot of the refrigerator we embedded a LakeShore Cernox CX1030 sensor into the sample-holder disk. The sensor is separated from the sample by a 0.3 mm sapphire substrate. Our experiments with this sample holder exhibited establishment of almost perfect temperature equilibrium between the refrigerator's cold flange and the sample with a time constant about 1 second.

3. Tests and operation

The early tests of the ^3He -refrigerator with the microscope head attached revealed that the total hold time for the base temperature of 300-400 mK can be as long as 20-24 hours. However while scanning

with all the electrical leads connected the hold time is reduced to about 3 hours. After this a new ^3He condensation cycle is required. In total it takes about 20 minutes to restore the base temperature.

We used a Pt-coated 1 μm period calibration grid from Veeco for the scanner calibration in topographic STM-mode with the Hall sensor replaced by an STM-tip. The latter test also revealed the high rigidity of the microscope frame and mechanical stability of the whole system under working conditions.

Imaging of magnetic structures was performed on an yttrium-iron-garnet (YIG) film employing a submicron GaAs/AlGaAs Hall probe. A layer of gold was deposited on the insulating sample surface prior to measurement in order to enable tunnel-tracking. Scanning was possible at speeds as fast as 300 $\mu\text{m/s}$ in ‘flying mode’ with the Hall probe just lifted out of tunnel contact. Labyrinth magnetic domain patterns in the YIG-film were captured in this flying mode (c.f. the high-speed unfiltered image presented in figure 2).

4. Conclusion

We have described the design, construction and performance of a novel SHPM for milliKelvin magnetic imaging. This instrument is capable of operation in magnetic fields up to 10T with sub-micron spatial resolution and minimum detectable fields $\geq 10\text{mG/Hz}^{1/2}$. Special measures were taken to provide good access to the sensors and the sample holder for their safe and quick replacement. The mechanically robust and rigid body of the detachable low-temperature microscope head makes it possible to exploit it over a wide temperature range.

Acknowledgements

The authors would like to thank Dr. A.N. Grigorenko from the University of Manchester for supplying the YIG sample. We gratefully acknowledge financial support from EPSRC in the UK (Grant No. EP/D034264/1).

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